

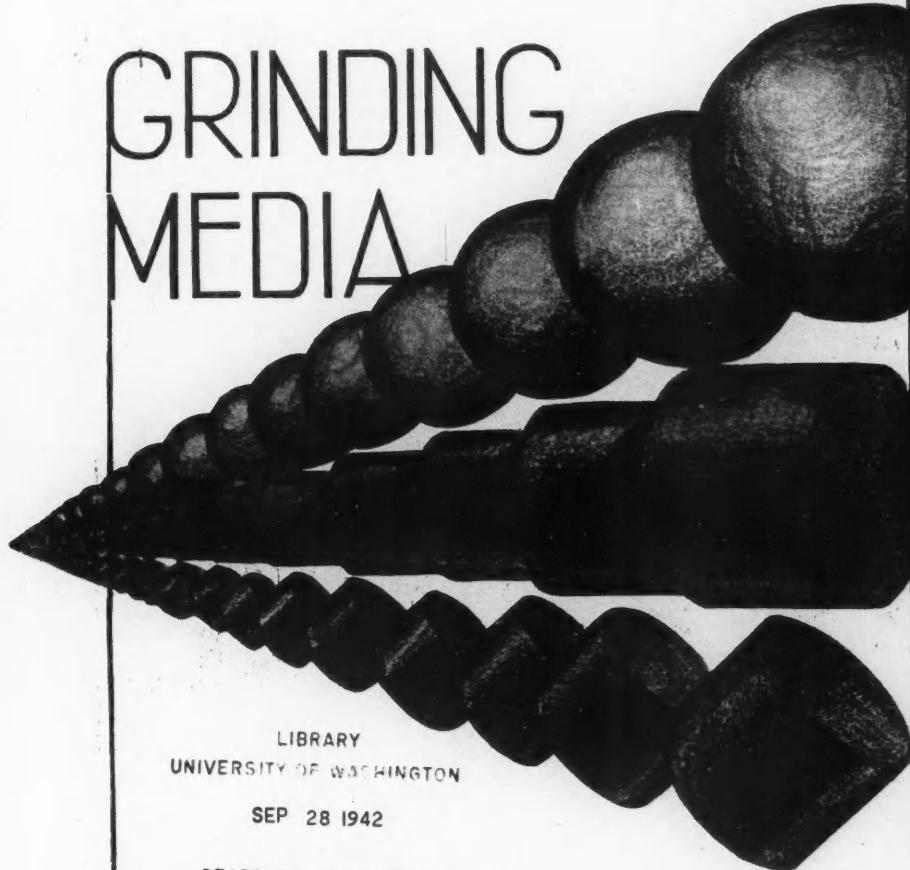
# CEMENT AND LIME MANUFACTURE

XV. No. 8

AUGUST 1942

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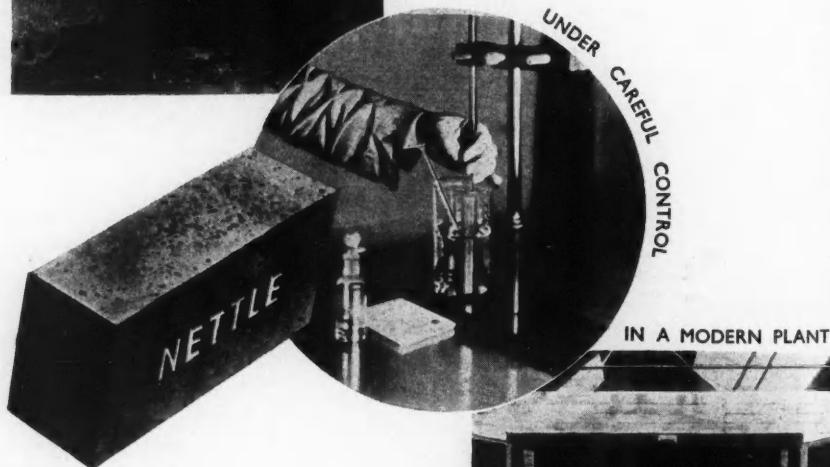
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## The Compound Mill for Wet Grinding.

AN article in the May, 1942, number of this journal dealt with the compound grinding mill, with special reference to its use under dry grinding conditions. The size of feed, the size of media, and other points to be considered in order to obtain the best results, were dealt with, but no reference was made to its use under wet grinding conditions. This application of the compound grinding mill is considered in the following.

Ball mills and tube mills were, and still are, used for wet-grinding cement raw materials, the former for preliminary grinding, and the latter for finish-grinding ; the ball mill is used because of the generally large size of feed that is in many instances still provided, and this large size feed is the result of the type of crusher used for finish-crushing the stone. The jaw crusher and the roll crusher have been, and are still, generally used for this duty, the former for breaking down the large pieces of stone, and the latter for finish-crushing the stone to an upper limiting dimension of, say, 1½in. or 1¾in. A secondary crusher of the roll type will only reduce over-size stone to the crusher setting, and a large proportion of its product will remain at or about the dimension of the setting ; this material, apart from its generally large size, will contain only a limited amount of "smalls," and the preliminary mill (ball mill) must be charged with large balls for dealing with the large-size material.

As the swing-hammer crusher, or hammer mill, developed, its product was found to have an entirely different characteristic from that of the roll crusher, and when the essential parts of the hammer mill are of suitable design and properly maintained, it is possible to obtain a product of which up to 90 per cent. will pass through an opening of 0·2in. Material of this size and of the classification produced makes an excellent feed for a mill of any type ; moreover, it is unnecessary to use balls of large size, and this at once does away with the need for the ball mill, and provides the opportunity for the compound mill. Further, an

economic or practical limit in size appears to have been reached in the construction of the ball mill, but no such limit appears likely with the compound mill; at any rate, compound mills have already been constructed to absorb several times the amount of power required by the ball mill, and the difficulties that have been met have been overcome.

The general principles of construction adopted in the compound mill, whether for wet or dry grinding, are the same, although some of the details differ. These mills are being used equally successfully for both wet and dry grinding; the construction, proportions and loading, and also the type and the size of media, are substantially the same, and the principal replacement parts may be of the same design for the same size mills.

Under dry grinding conditions the media must be suitable for producing the smallest practicable particle size, and the whole of the product of the mill must have the largest possible amount of surface area per pound weight. Under wet grinding conditions the upper limiting size of particle is determined by the behaviour of the particles under the temperature conditions of the kiln, but large quantities of very small particles might prove a nuisance and be the cause of considerable loss. It is possible that the largest particle size of most raw materials might be, say, 0.025in., and be quite suitable; a small particle size of, say, 0.005in. might also be satisfactory, and all particles between these dimensions would possibly satisfy chemical requirements—the actual size of particle can only be determined in practice.

In considering the size of media necessary for efficient duty under dry grinding conditions, the rule  $D = 20d + \frac{1}{4}$  in. has been found satisfactory when  $D$  is the mean diameter of the media and  $d$  the mean diameter of the particle of grit to be ground easily in the various chambers; this rule is a compromise between the ideal and practical conditions, and is satisfactory when the number of chambers is kept as small as practicable and when the wear of the media is not abnormal. The wastage by wear is greatest when dealing with a coarse product, although the wastage per ton ground is the lowest; the wastage by wear per ton of product increases with a finer product.

Under wet grinding conditions the wastage by wear would be higher (actually several times higher) than under dry grinding conditions, but most of the loss may possibly be associated with, or be the result of, oxidation.

The larger particle size in the finished product of the wet mill and the higher rate of wastage of media together appear to call for a re-examination of the rule for the size of media.

The dash line in Fig. 1 (which is here reproduced from the earlier article) makes provision for wear and wastage at the feed end of the dry mill. A similar provision must be made for the wet mill, and the basis for the size of media for the wet mill may be considered on the following lines.

The "curve of media diameter" for the dry mill indicates that a mean particle of 0.06in. (an assumed mean figure for the last chamber in the wet mill)

will call for a mean size of  $1\frac{1}{2}$  in. for the media for this chamber, and the range of size may be, say, 2 in. to  $1\frac{1}{2}$  in., but, owing to probable increased wastage, an addition of  $\frac{1}{4}$  in. to  $\frac{1}{2}$  in. may be made to all media used in this chamber. This size and range could be continued in this chamber until the efficiency falls or until the small media become troublesome or too small for the duty.

Owing to rapid wear and wastage in a wet mill, too much care cannot be taken in obtaining the most suitable media, or the media that give the longest

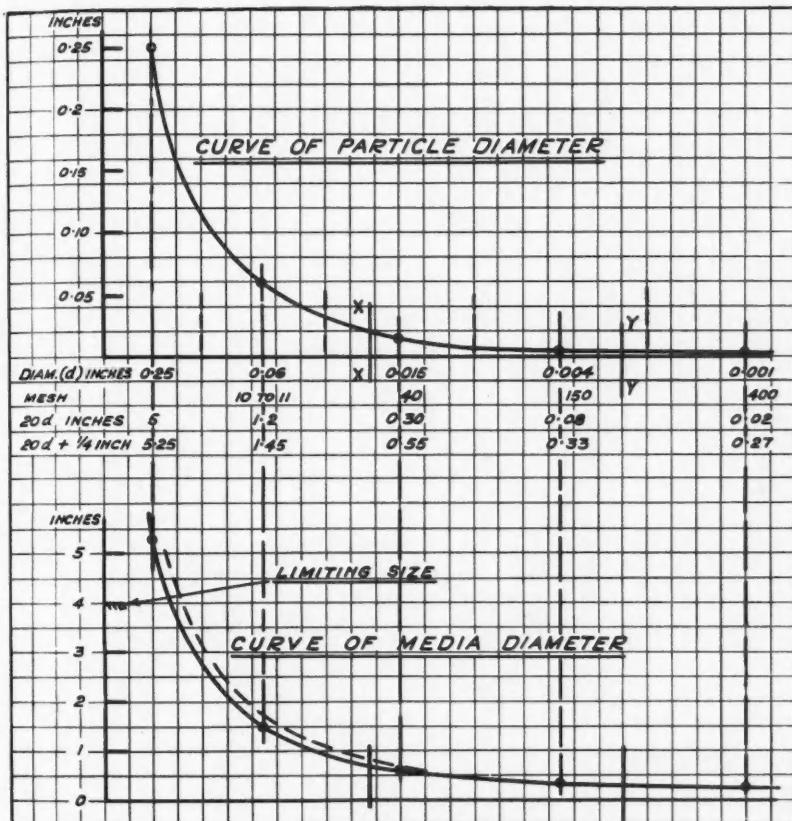


Fig. 1.—Curves of Particle-Media Ratio.

service generally. High-carbon hardened forged steel should prove the best for the feed end of the mill and for all media down to, say,  $1\frac{1}{2}$  in. diameter, but the smaller media must be treated experimentally, and final selection must be based upon actual experience. Although wear of large media will produce small media, it will not produce enough, and an amount of make-up will always be required for the finishing end of the mill.

It is not unusual for rake, or other type, classifiers to be used in conjunction with wet mills of considerable dimensions, and such classifiers will have some influence upon the internal arrangement of the mill and also on the size of media. The use of a classifier obviates the need of using any small media, and reduces the need for multiple chambers. The adoption of a classifier calls for additional maintenance, which may be considerable, but the reduced wear and cost of media appear to fully justify its use; moreover, the output of the mill will be higher.

When a classifier of any kind is adopted, media of larger size can be used all through the length of the mill, any over-size grit being caught at the classifier and automatically returned to the mill for further grinding. This procedure reduces the amount of excess grinding of the smallest particles, as these are removed from the mill as soon as they reach or approach the limiting dimension, and the product of the mill will thus be all, or nearly all, "smalls," but no, or very little, "fines."

The internal arrangement of a wet mill may be in substantial agreement with that of a dry mill, with the following exceptions.

**MEDIA AND CHAMBERS.**—As already stated, the particle size of the finished product under wet-grinding conditions would be larger than that obtained from dry grinding, and there would be no need for any small, or very small, media apart from those which had reached the worn-out condition and have not been removed from the mill. As no small media will be in use, the number of chambers will be less than for dry grinding. The length of the first chamber would be in agreement with that for dry grinding, say, 30 per cent. of the net length of the mill; the other chambers would make up the total length of the mill.

**DIAPHRAGMS.**—There will be no need for a lifter diaphragm, so all the diaphragms will be of the plain or plate type. For normal duty the slotted openings in the first diaphragm would be, say, 2 to 2.5 mm. wide; this dimension will establish an upper limit to particle size at this point, it being assumed that the residue reduction capacity of the media beyond this point would result in the fineness or residue required. The width of the slots in the other diaphragms might be in substantial agreement with that of the first diaphragm.

In dry grinding a lifter diaphragm is considered necessary at the first position, and this controls the amount of grit retained in the first chamber, and also establishes a "head" for the second and other chambers to ensure the flow of cement through the mill. This provision is not necessary in wet grinding; in fact, provision must be made for restricting the flow of slurry or moisture. This restriction is usually effected by having a blank margin outside the slotted area in the last diaphragm, the radial depth of this blank area being about equal to the depth of the charge. Alternatively, and possibly preferably, the last diaphragm would be slotted over the whole area in the same way as the others, and an additional annular diaphragm would be arranged as a weir a few inches from, and on the down-stream side of, the last slotted diaphragm; this proves a very effective control, and it also prevents the mill from emptying itself when it is not working.

The whole of the mill discharge would flow over this weir, and this should largely prevent any over-size pieces of stone from leaving the mill.

A blank diaphragm, or something equivalent, is necessary when the mill is arranged with a peripheral discharge, but a diaphragm-with-lifters or something equivalent is necessary for a trunnion discharge.

Serious scouring or cutting action is possible under the conditions obtaining in wet grinding, and every effort should be made in design and during assembly to prevent leakage between the various chambers and also between the lining and the mill shell. The design of the linings should result in definite lifting action when they are new, and also prevent slipping when they approach the worn-out condition.

**POWER.**—Relative and approximate data in connection with the net power absorbed by the charge in the mill under wet and dry conditions are given in Table I.

TABLE I

Material	Weight (lb. per cu. ft.) Solid	No. of pieces or balls
Forged steel ..	490	265 to 275
Cast iron ..	450	245 to 255
Limestone ..	160 to 170	88 to 94
Cement grit ..	—	88 to 92
Water .. ..	62.5	—
Slurry .. ..	88 to 94 (40% moisture)	—

(The figures are typical rather than precise.)

Amplifying and qualifying data are as follows. As the bulk and weight of the media are the same under both wet and dry conditions, and as the weight of the slurry or cement grit may be substantially the same, there should be only a small difference in the net power required whether the mill is running wet or dry. The horse-power required per ton of material ground in the two processes will thus be largely a factor of the difference in the resistance-to-grinding of the material and the fineness of the finished product, assuming that the classification of the media is substantially correct. Under these conditions the total power required by the mill will be determined or influenced by the following.

(1) Diameter of mill.—The horse-power required per ton of media in a large mill, at the same basic speed, will be rather greater than that required for a smaller mill owing to the increased height of lift of the charge mass.

(2) Total weight of media.—The reason for this difference will at once be obvious; under usual conditions of operation the approximate power required by a mill will vary proportionately with the total weight of charge.

(3) Volume-ratio of charge.—The horse-power per ton required for a 20 per cent. charge volume will be rather higher than for a 40 per cent. charge volume. It may be difficult to observe the difference between, say, 20 per cent. and 25 per

cent., or between 35 per cent. and 40 per cent., owing to the several influencing factors and the difficulty in obtaining precise readings.

(4) Speed of mill.—Within the usual limits of speed adopted in practice, the power absorbed by any mill will be in direct proportion to the speed, providing other conditions are comparable. The apparent best speed is, say, three-quarters of the critical speed. The critical speed in revolutions per minute is that speed at which particles of positive weight may begin to cling or adhere to the mill lining, and this speed is determined by the formula : Critical speed =  $\frac{54.19}{\sqrt{R}}$ , where  $R$  is the radius of the inside of the mill in feet.

The best speed named (75 per cent. of the critical speed) may be suitable for a charge volume of 30 per cent., and any increase in charge volume will call for an increase in speed, or vice versa ; a charge volume greater than 30 per cent. should only be adopted under wet-grinding conditions.

The theoretical speed in relation to charge volume is discussed very fully by E. W. Davis in *Bulletin No. 146/1919* of the American Institute of Mining Engineers ; but all the speeds arrived at by the author are much lower than those arrived at by the formula given above.

#### General.

The weight and crushing capacity of balls varies as the cube of their diameters, and for this reason it should be an advantage to use the largest balls practicable under wet conditions, and especially when a classifier of any type is used ; the ultimate wastage of ball media will also be less.

In reviewing the data, it appears that there is much to be said in favour of the compound grinding mill for wet grinding, whatever the material, assuming that a suitable size and classification of feed can be obtained. It further appears that there is much to be said in favour of adopting a classifier to work in conjunction with the mill, provided the mill is large enough to justify the cost.

The classifier not only classifies the ground or partly ground material, but it enables a simpler mill to be used ; it eliminates the need for small media, and it avoids excess grinding of the material by removing it from the mill as soon as it reaches the required size ; and, as classification is the last stage in the process, the classifier definitely avoids the risk of nibs being passed out with the finished product.

[In considering the foregoing, it is desirable to do so in conjunction with the article, already referred to, in the May number of this journal.]

#### The Edison Cement Works.

It is reported from the United States that the cement works established in 1898 near Phillipsburg, New Jersey, by the late Thomas A. Edison is to close down, due to the high cost of hauling limestone a distance of twelve miles. The works, now owned by the Edison Cement Corporation, has a capacity of about 350,000 tons a year.

## Chemical Analysis of Portland Cement.

### NEW METHODS OF DETERMINATION.

ALTERNATIVE methods for the determination of sodium oxide, potassium oxide, and sulphur occurring as sulphide, have been recommended by the Committee on Cement of the American Society for Testing Materials. These are as follows.

#### SODIUM OXIDE AND POTASSIUM OXIDE.

##### Reagents.

(a) *Zinc Uranyl Acetate Reagent*.—Shake a mixture of 20 gr. of uranyl acetate ( $\text{UO}_2(\text{CH}_3\text{CO}_2)_2 \cdot 2\text{H}_2\text{O}$ ), 60 gr. of zinc acetate ( $\text{Zn}(\text{CH}_3\text{CO}_2)_2 \cdot 2\text{H}_2\text{O}$ ), 5 ml. of glacial acetic acid, and 175 ml. of warm water continuously for about one hour or at intervals for a few hours. If the solution is clear, add a little NaCl or sodium zinc uranyl acetate ( $\text{NaZn}(\text{UO}_2)_3(\text{CH}_3\text{CO}_2)_6 \cdot 6\text{H}_2\text{O}$ ) to the solution and shake it again. It is essential that the reagent is saturated with respect to sodium zinc uranyl acetate at the temperature at which the reagent is used. Allow the reagent to stand until its temperature is constant. Filter small portions of it as required, keeping the temperature as uniform as practicable in all operations involving the preparation of the reagent and alcoholic wash and the precipitation, filtration, and washing of the sodium zinc uranyl acetate.

(b) *Alcoholic Wash*.—Add some sodium zinc uranyl acetate to ethanol which contains acetic acid (1 per cent.), and shake the solution continuously for about one hour or at intervals for a few hours. Filter portions of the wash as required (see paragraph (a) in regard to temperature). The ethanol may be absolute or 95 per cent. and may be pure or denatured according to Formula No. 2b or 3a of the U.S. Bureau of Internal Revenue.

(c) *Chloroplatinic Acid*.—Dissolve 5 gr. of  $\text{H}_2\text{PtCl}_6 \cdot 6\text{H}_2\text{O}$  in 95 ml. of water and 5 ml. HCl specific gravity 1.19.

(d) *Gladding Wash*.—Dissolve 100 gr. of  $\text{NH}_4\text{Cl}$  in 500 ml. of water and add to the solution an amount of  $\text{K}_2\text{PtCl}_6$  that is more than sufficient to make a saturated solution (about 1 gr.). Shake the mixture frequently for six to eight hours, allow it to stand at least twelve hours, and filter. The residue may be used for the preparation of a new supply of the wash.

##### Procedure.

(a) *Preparation of Solution*.—To 2 gr. of cement in a platinum dish, preferably of the flat-bottom type, add 5 ml. of cool water, stir the mixture with a platinum rod, add carefully 10 ml. of  $\text{NHO}_3$  (1 : 1), and continue stirring until the cement is completely decomposed. Add 10 ml. of  $\text{HClO}_4$  (60 per cent.) and 25 ml. of HF (48 per cent.) (Note 1) to the solution and mix it thoroughly with the rod. Remove the rod and rinse it with water. Evaporate the solution to dryness or fumes of  $\text{HClO}_4$  (Note 2). Cool the dish, wash down the side with a little hot water, and repeat the evaporation. Drive off the excess  $\text{HClO}_4$  by cautiously moving a flame under the dish or increasing the temperature of a radiator, taking care not to heat the dish to redness. Cover the dish with a watch glass of fused silica or heat-resistant glass and convert the perchlorates to chlorides by using

stronger heat but still avoiding red heat. The residue fuses to a brown mass and may glow from an exothermic reaction. The reaction may be carried out by heating a small part of the residue at a time and applying no more heat than is necessary to maintain the reaction. After the completion of the reaction, continue the heating for one or two minutes below red heat. Cool the dish and watch glass (Note 3). Rinse the watch glass into a beaker. Add a little water to the dish, let the residue stand a few minutes to soften, and grind it with a glass mushroom or pestle to a smooth paste (Note 4). Wash the contents into the beaker and dilute them to 40 to 50 ml. Boil the contents for 15 minutes, keeping the volume between 25 and 40 ml. Add a little macerated filter pulp to the contents, and filter the solution through a filter paper of medium or fine porosity into a 100-ml. volumetric flask which contains sufficient HCl to acidify the filtrate (Note 5). Wash the filter paper and residue at least ten times until the volume is nearly 100 ml. Cool the filtrate (Note 6), dilute to the mark, and mix thoroughly.

(b) *Determination of Na<sub>2</sub>O.*—Transfer a 20-ml. portion of the solution [paragraph (a)] to a small vessel and evaporate it to dryness (Note 7). Cool the vessel, take the residue up in 1 ml. of water, add 15 ml. of the zinc uranyl acetate reagent while stirring, and allow the contents to stand for 30 to 60 minutes (Note 8) with frequent stirring. Filter the solution through a weighed (Note 9) 15-ml. fritted-glass filtering crucible of medium porosity or a Gooch crucible with suction. Transfer the precipitate to the crucible and scrub and rinse the beaker with a rubber "policeman," using small quantities of the zinc uranyl acetate reagent. Wash the precipitate six times with 2-ml. to 3-ml. portions of the alcoholic wash (Note 10), being careful to wash down the side of the crucible, and three times with 4-ml. to 5-ml. portions of anhydrous ether. Draw air through the crucible until the odour of ether disappears, wipe the outside of the crucible with a damp cloth, place the crucible in a desiccator for one hour (or 15 minutes if *in vacuo*), and weigh it. Calculate the percentage of Na<sub>2</sub>O from the weight of sodium zinc uranyl acetate as follows:

$$\text{Na}_2\text{O} \text{ per cent.} = W \times 5.0375$$

where  $W$  = weight of  $\text{NaZn}(\text{UO}_2)_3(\text{CH}_3\text{CO}_2)_9 \cdot 6\text{H}_2\text{O}$ , and

$$5.0375 = \text{ratio of Na}_2\text{O to NaZn}(\text{UO}_2)_3(\text{CH}_3\text{CO}_2)_9 \cdot 6\text{H}_2\text{O} \text{ divided by } 0.4 \text{ and multiplied by 100, or } \frac{0.02015}{0.4} \times 100.$$

(c) *Determination of K<sub>2</sub>O.*—Transfer a 75-ml. portion of the solution [paragraph (a)] to a vessel and evaporate the solution to about 5 ml. Add 5 ml. of the chloroplatinic acid to the solution and continue the evaporation on a water bath until the liquid solidifies to a soft mass upon cooling (Note 11). Add 30 ml. of ethanol (85 per cent.) to the vessel and grind the residue finely with a glass mushroom. Allow the contents to stand for about one hour with frequent stirring and filter through a weighed 15-ml. fritted-glass filtering crucible of medium porosity with suction. Scrub and rinse the vessel with as little ethanol (85 per cent.) as possible. Wash the crucible and residue five times with 3-ml. to 5-ml. portions of ethanol (85 per cent.), then six times with the Gladding wash with the suction shut off and the residue churned up thoroughly with a fine

stream of the wash each time (Note 12), and finally six times with ethanol (85 per cent.) with care taken to wash down the side of the crucible. Dry the crucible at 105 deg. C. to 110 deg. C. for at least two hours or 130 deg. C. to 135 deg. C. for at least 30 minutes, cool in a desiccator, and weigh. Calculate the percentage of K<sub>2</sub>O from the weight of K<sub>2</sub>PtCl<sub>6</sub> as follows:

$$\text{K}_2\text{O} \text{ per cent.} = W \times 12.92$$

where  $W$  = weight of K<sub>2</sub>PtCl<sub>6</sub>, and 12.92 = ratio of K<sub>2</sub>O to K<sub>2</sub>PtCl<sub>6</sub> divided by 1.5 and multiplied by by 100, or  $\frac{0.1938}{1.5} \times 100$ .

NOTE 1.—Care should be taken that the acid does not carry any wax from its wax bottle, as organic matter in the presence of HClO<sub>4</sub> at a high temperature may cause an explosion.

NOTE 2.—The evaporation requires care as the contents have a tendency to bump or spatter, especially when the dish is in contact with a flame or a hot plate at a high temperature. The best way to handle the evaporation depends on the equipment available. If practicable, the dish may be left on a water bath or a hot plate at a low temperature overnight. If the hot plate is too hot, a mat of asbestos may be used under the dish. A radiator may be used in which the solution is heated by infra-red reflector drying lamps placed below or above the dish, or both. If a gas-fired radiator is used, its bottom should be closed so that the dish is heated by radiation rather than by conduction. A radiator of this type may be constructed out of stock 2-in. plumbing fittings: Place a 3in. support ring over a close nipple and screw a cap and coupling on the nipple until they come together and hold the ring firmly. As the iron is thick, a burner of the Meker type may be used for heating. If a similar radiator is made of thin copper or galvanised iron, a Bunsen burner may be used. The contact of platinum with iron should be avoided. If the residue is allowed to stand overnight after the second evaporation, it may be placed in an oven or desiccator to prevent delay the next day due to absorption of moisture.

NOTE 3.—As ammonium chloroplatinate is like potassium chloroplatinate in being insoluble in ethanol, all following steps involving the determination of potassium oxide should be carried out in an atmosphere free of ammonia and ammonium salts.

NOTE 4.—The residue should be thoroughly ground to ensure the complete extraction of sodium and potassium chlorides. If the bottom of the dish is round and the grinding cannot be done satisfactorily, the residue may be transferred to a casserole. Decant most of the water into the beaker and grind the residue.

NOTE 5.—One or two drops of acid are usually sufficient. Filtration under suction may be employed. A convenient arrangement is to use a large bell glass, the edge of which is greased and in contact with a piece of plate glass. The top should have a 1in. to 2in. opening fitted with a two-hole rubber stopper. Insert a funnel and a glass tubing through the stopper. Connect the tubing with a T-shape connecting tube and an aspirator or vacuum pump. The connecting tube enables one to release the vacuum without turning off the aspirator or pump. The end of the tubing within the bell glass should be curved toward the side and fitted with a rubber tubing which extends to the bottom of the bell glass. This prevents spoiling work by the back rush of air or water. The arrangement makes it possible to filter with suction directly into vessels of many types and sizes.

NOTE 6.—It is not necessary to cool to any particular temperature so long as the flask and pipettes used in the following steps are calibrated at the same temperature and the temperature of the solution is close to the temperature of calibration.

NOTE 7.—The first part of the evaporation may be done over a burner or hot plate with a stream of air directed on the surface and finished on a water bath or in a drying oven at 105 deg. C.

NOTE 8.—The solution should stand at least one hour when the content of Na<sub>2</sub>O is low (less than 0.2 per cent.).

NOTE 9.—Wash, dry, and weigh the empty crucible just as it is done when filtered sodium zinc uranyl acetate is present.

NOTE 10.—When the reagent and alcoholic wash are mixed and allowed to stand, a white precipitate results which does not dissolve in an excess of the wash. The washing with the alcoholic wash should be done rapidly.

NOTE 11.—The content of K<sub>2</sub>O is assumed to be less than 1.5 per cent. Five millilitres of the chloroplatinic acid are about twice the amount necessary to precipitate the potassium in cement having a K<sub>2</sub>O content of 1.5 per cent. If the content is much greater than 1.5

per cent., the amount of the acid should be increased in proportion. The liquid may contain so much calcium chloride that toward the end of the evaporation most of the calcium chloride and some of the  $K_2PtCl_6$  may crystallise out, giving slush instead of a syrupy solution. The last stage of the evaporation requires care, and it may be necessary to cool and re-evaporate a few times. If the evaporation is too prolonged, the chloroplatinic acid may be partially decomposed or the solidified mass may be hard to break up in ethanol.

NOTE 12.—The  $K_2PtCl_6$  is contaminated with considerable material which is insoluble in ethanol but soluble in the Gladding wash. It is thus essential that the washing with the Gladding wash be done thoroughly.

### SULPHUR AS SULPHIDE.

#### Apparatus.

*Gas Generating Flask.*—Connect a dry 500-ml. boiling flask with a long-stem separatory funnel and a small connecting bulb by means of a rubber stopper. Bend the stem of the funnel so that it will not interfere with the connecting bulb, adjust the stem so that the lower end is close to the bottom of the flask, and connect the opening of the funnel with a source of compressed air. Connect the bulb with an L-shape glass tube and a straight glass tube about 20 cm. in length. Insert the straight glass tube in a tall 400-ml. beaker. A three-neck Woulff bottle with a long glass tubing in the middle opening, placed between the source of compressed air and the funnel, is a convenient aid in the regulation of the air flow. If the air contains  $H_2S$  or  $SO_2$ , a solution of lead acetate or other suitable absorbent is used in the bottle. Rubber used in the apparatus shall be of a pure-gum grade low in sulphur and shall be cleaned with warm HCl.

#### Reagents.

(a) *Starch Solution.*—To 100 ml. of boiling water, add a cool suspension of 1 gr. of soluble starch in 5 ml. of water and cool. Add a cool solution of 1 gr. of NaOH in 10 ml. of water, then 3 gr. of KI, and mix thoroughly.

(b) *Potassium Permanganate Solution (approximately 0.03 N).*—Prepare a solution of  $KMnO_4$  on the basis of 0.94 gr. per litre. The solution should not be filtered through any filter containing organic matter. It is most convenient to siphon off clear solution without disturbing the sediment on the bottom of the bottle. Standardise the solution against about 0.15 gr. of sodium oxalate oxidimetric standard furnished by the National Bureau of Standards (standard sample No. 40) according to the directions furnished with the sodium oxalate.

(c) *Sodium Thiosulphate Solution (approximately 0.03 N).*—Prepare a solution of  $Na_2S_2O_3$  on the basis of 7.4 gr. of  $Na_2S_2O_3 \cdot 5H_2O$  per litre.

(d) *Potassium Iodate Solution (approximately 0.03 N).*—Prepare a solution of  $KIO_3$  and KI on the basis of 1.12 gr. of  $KIO_3$  and 12 gr. of KI per litre. Standardise the solution as follows: To a cool solution of 1 gr. of KI in 300 ml. of water and 10 ml. of HCl (sp. gr. 1.19) in a 500-ml. flask, add about 25 ml. of the  $KMnO_4$  solution, swirl the solution gently, stopper the flask, and let stand for 5 minutes. Titrate the liberated iodine with the thiosulphate solution until the colour nearly fades. Add 2 ml. of the starch solution, continue the titration until the blue colour is destroyed, and back-titrate with the  $KMnO_4$  solution (Paragraph (b)) until the blue colour just reappears. Repeat the titration with the  $KIO_3$  solution substituted for the  $KMnO_4$  solution. Calculate the value of

the standard solution of  $KIO_3$  in terms of grammes of sulphur per millilitre as follows :—

$$\text{Value of } KIO_3 \text{ solution} = \frac{A \times C \times F \times 0.2392}{B \times D \times E}$$

where

$A$  = weight in grammes of  $Na_2C_2O_4$  used in the standardisation of the  $KMnO_4$  solution,

$B$  = volume in millilitres of  $KMnO_4$  solution required by  $A$ ,

$C$  = volume in millilitres of  $KMnO_4$  solution used in the standardisation of the  $KIO_3$  solution,

$D$  = volume in millilitres of  $Na_2S_2O_3$  solution required by  $C$ ,

$E$  = volume in millilitres of  $KIO_3$  solution used in the standardisation of the  $KIO_3$  solution, and

$F$  = volume in millilitres of  $Na_2S_2O_3$  solution required by  $E$ .

NOTE.—A normal solution of  $KMnO_4$  or  $KIO_3$  is equivalent to 0.06701 gr. of  $Na_2C_2O_4$  or 0.01603 gr. of sulphur. The number 0.2392 is obtained by dividing 0.01603 by 0.06701. The  $KIO_3$  and  $KMnO_4$  solutions should be standardised frequently but, as the  $Na_2S_2O_3$  solution is more stable, the  $KIO_3$  solution may sometimes be standardised against the  $Na_2S_2O_3$  solution alone and without the last values of  $A, B, C$ , and  $D$  being changed.

(e) *Stannous Chloride Solution*.—To 10 gr. of  $SnCl_2 \cdot 2H_2O$  in a small flask add 7 ml. of diluted HCl (1 : 1), warm the mixture gently until the salt is dissolved, cool the solution, and add 95 ml. of water. This solution should be made up as needed, as the salt tends to hydrolyse.

(f) *Ammoniacal Zinc Sulphate Solution*.—Dissolve 50 gr. of  $ZnSO_4 \cdot 7H_2O$  in 150 ml. of water and 350 ml. of  $NH_4OH$  (sp. gr. 0.90). Filter the solution after allowing it to stand at least 24 hours.

(g) *Ammoniacal Cadmium Chloride Solution*.—Dissolve 15 gr. of  $CdCl_2 \cdot 2H_2O$  in 150 ml. of water and 350 ml. of  $NH_4OH$  (sp. gr. 0.90). Filter the solution after allowing it to stand at least 24 hours.

#### Procedure.

Place 15 ml. of the ammoniacal  $ZnSO_4$  solution (Note 1) and 285 ml. of water in the beaker. Put 5 gr. of the sample (Note 2) and 10 ml. of water in the flask and shake the flask gently to wet and disperse the cement completely. This step and the following one should be performed rapidly to prevent the setting of the cement. Connect the flask with the funnel and bulb. Add 25 ml. of the  $SnCl_2$  solution through the funnel and shake the flask. Add 100 ml. of diluted HCl (1 : 3) through the funnel and shake the flask. During these shakings, keep the funnel closed and the delivery tube in the ammoniacal  $ZnSO_4$  solution. Connect the funnel with the source of compressed air, open the funnel, start a slow stream of air, and heat the flask and contents slowly to boiling. Continue the boiling gently for 5 or 6 minutes, cut off the heat, and continue the passage of air for 3 or 4 minutes. Disconnect the delivery tube and leave it in the solution for use as a stirrer. Cool the solution to 20 to 30 deg. C. (Note 3), add 2 ml. of the starch solution and 40 ml. of diluted HCl (1 : 1), and titrate immediately with

the  $\text{KIO}_3$  solution until a permanent blue colour is obtained (Note 4). Calculate the percentage of sulphur (Note 5) as follows :

$$\text{sulphur, per cent.} = \frac{G \times H}{W} \times 100$$

where

$G$  = sulphur equivalent of the  $\text{KIO}_3$  solution in grammes per millilitre,

$H$  = volume in millilitres of  $\text{KIO}_3$  solution used in the titration, and

$W$  = weight in grammes of sample.

NOTE 1.—In general the  $\text{ZnSO}_4$  solution is preferable to the  $\text{CdCl}_2$  solution because  $\text{ZnSO}_4$  is more soluble in  $\text{NH}_4\text{OH}$  than is  $\text{CdCl}_2$ . The  $\text{CdCl}_2$  solution may be used when there is doubt as to the presence of a trace of sulphide sulphur, as the yellow  $\text{CdS}$  facilitates the detection of a trace.

NOTE 2.—If the content of sulphur exceeds 0.20 or 0.25 per cent., a smaller sample should be used so that the titration with the  $\text{KIO}_3$  solution will not exceed 25 ml.

NOTE 3.—The cooling is important as the end point is indistinct in a warm solution. A part of the  $\text{NH}_4\text{OH}$  is lost during the distillation and the remaining  $\text{NH}_4\text{OH}$  reacts with acid, raising the temperature of the solution a few degrees without rendering the end point uncertain.

NOTE 4.—If the content of sulphur is appreciable but not approximately known in advance the result may be low due to the loss of  $\text{H}_2\text{S}$  during a slow titration. In such a case the determination should be repeated with the titration carried out more rapidly.

NOTE 5.—Sulphites, thiosulphates, and other compounds intermediate between sulphides and sulphates are assumed to be absent. If such compounds are present, they may cause an error in the determination.



## Automatic Controls for Kilns and Tube Mills.

AT one of the works of the Pennsylvania-Dixie Cement Corporation, eight 100 ft. kilns have been replaced by four kilns, two of which are 10 ft. in diameter by 140 ft. long, and the other two 10 ft. in diameter by 136 ft. long. Each kiln is connected to a waste-heat boiler, and produces from 215 to 235 tons a day. The kilns are insulated for two-thirds of their length and lined for their full length.

Each kiln has a separate mill to grind the coal, dry it, and inject the coal and primary air into the kiln, all in one operation. The mills are 5 ft. by 6 ft. air-swept tube mills operated under suction, and have an automatic feature to keep the coal level inside the mill to the point of optimum grinding efficiency. Each mill has an integral drive gear and is driven from a 50-h.p. motor through a flexible coupling. The loading is 9,120 lb. of forged steel balls, 20 per cent. 2 in. and 80 per cent. 1½ in. New balls are added without stopping the mill by dropping them into an opening at the coal feeder. The mills turn at 30 r.p.m. and are rated at 5,000 lb. of coal (and moisture) each per hour at a fineness of 88 to 90 per cent. through 200-mesh.

The mills are on the floor below the kiln room, and the feeders and the suction fans are on the kiln firing floor. Coal is conveyed into steel bins, each holding enough coal of 1 in. top size sufficient for an 18-hour run and equipped with bin-level indicators. The bins have been "belled-out" near their bottoms, which is an effective means of preventing arching and stimulating a steady flow to the feeder. Each table feeder is driven by a ½ h.p. variable-speed induction motor, and each air fan is direct-connected to a 15-h.p. motor.

Clinker is burned at about 2,800 deg. F. and about 30 per cent. of the combustion air is preheated primary air. Most of the secondary air is preheated to about 1,000 deg. F., coming from the air-quenching clinker coolers. Air for drying the coal while it is ground in the mill is taken from the kiln hood and is drawn through the tube mill and injected, with the pulverised coal, into the kiln burner pipe. However, more primary air is needed than can be put through the tube mill. Pulverised coal from the tube mill discharge is taken up a vertical 12 in. diameter pipe to the kiln floor, and only about one-third of the primary air is needed to lift the fine coal. This volume of air is kept at a constant figure in order not to raise particles that are too coarse. In addition, part of the vertical pipe is enlarged to 22 in. diameter to reduce the speed of the gases so that coarse coal particles drop back into the mill. Hot air drawn from the kiln hood is divided into two branch lines. Ahead of the division is a temper damper at the kiln hood and a cyclone where clinker dust is settled out. One branch line carries heated air, after tempering, into the vertical pipe through which coal drops from the feeder table down to the tube mill. The junction of the coal and air is just below the feeder. The remainder of the primary air goes through the second, or by-pass, line to join the primary-coal mix from the tube mill as it enters the fan to be forced into the kiln. In order to take care of variations in

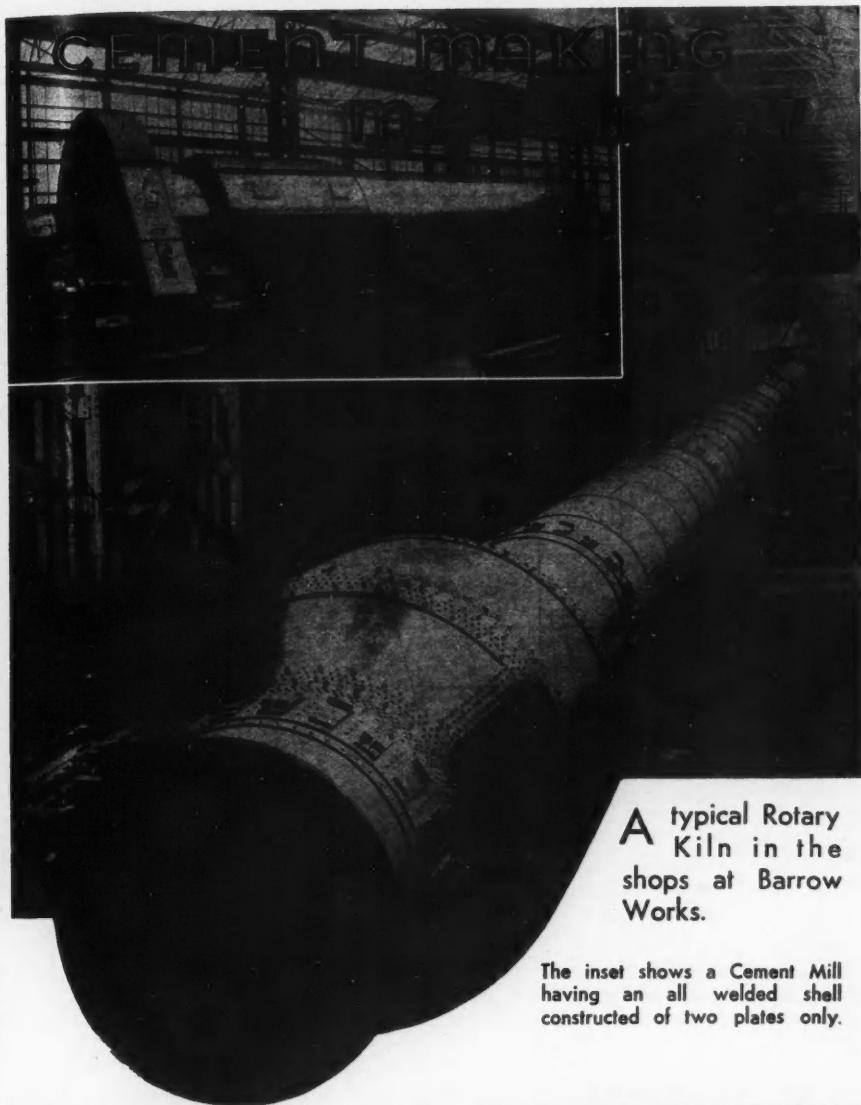
the moisture content of the coal, the temperature of the air entering the mill is varied by changing the quantity of coal air admitted by the tempering damper. The damper is automatically actuated so as to maintain the temperature of the coal-laden air leaving the mill constantly at 150 deg. F. Temperatures of the heated air entering the tube mills are not a consideration. They may be as low as 200 deg. F. or as high as 700 or 800 deg. F. depending upon the content of the coal. The temperature of the extra primary air introduced at the fan is tempered manually by a damper to 250 deg. F.

An automatic mill level control regulates the flow of coal into the grinding mills within close limits for best grinding efficiency and the maximum production of fines. Variation of the dielectric media (air and coal dust) between the two plates of a condenser, which consists of a rod extending into the mill and the mill itself, causes a variation of a high-frequency current which is generated in an electron tube and passes over a bridge circuit. A detector tube rectifies this high-frequency current, which may either be passed over sensitive relays or may be amplified in another tube. The variations of this rectified signal current are utilised to change the speed of the coal-feeder motor. The motor drive on the feeder is capable of these changes within reasonable limits, but should a greater change be necessary the knives on the table feeders can be adjusted manually.

Balls are added into a mill through the coal-feeder spout when indicated as necessary by the mill-drive ammeter or by coal-fineness tests. Coal finenesses are ascertained daily. Samples are withdrawn from the burner pipes through a valve fitting through which a hollow brass pipe is pushed into the coal stream to predetermined levels so as to obtain a proper cross sectional sample. This pipe is connected to a flexible hose through which an ordinary house vacuum cleaner draws a sample into its bag. Coal is injected into the kilns through 10 in. diameter water-cooled burner pipes of plain steel that project inside the hoods to within a foot of the end of the kiln. Inside the kiln hood the pipe is protected by a 2 in. covering of insulating cement.

Clinker from each discharges over a 5 ft. by 20 ft. 6 in. inclined grate clinker cooler where it is quenched by air as it flows over the grates. Two separate air lines from a single fan introduce the air through the primary, or quenching, section and through the secondary section of the cooler, which are separated by a partition. That air preheated by the clinker in passing through the primary end of the cooler constitutes the secondary air needed by the kiln for combustion. The clinker discharged from the cooler is at air temperature, or slightly more, and is conveyed direct to storage.

Clinker is burned in the new kilns with 10 per cent. excess air and a draught of 0.05 in. of water measured inside the kiln hood. The draught is kept constant by an automatic control which operates the louvres at the kiln stack fans to control the volume of gases through the kiln. The combination of a constant rate of feed of pulverised coal and automatic regulation of the flow of air keeps constant burning conditions inside the kiln. Each kiln has an optical pyrometer focussed through a peep-hole in the kiln hood upon the kiln lining. Hot zone temperatures



A typical Rotary Kiln in the shops at Barrow Works.

The inset shows a Cement Mill having an all welded shell constructed of two plates only.

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as measured by the instrument are continuously recorded on a potentiometer, along with the kiln revolutions and the rate of feed of raw material into the kiln. The pyrometers are a means of having a continuous temperature record and indicate at a glance when adjustments are needed. The volume of air into the kilns is never changed. If kiln temperatures vary sufficiently to require a change, the kiln speed is adjusted at the panel-board by resetting the kiln-drive rheostat, and the kiln-feed-drive rheostat automatically changes with it.

Another continuous recording chart shows the dust chamber temperature and the gas temperature just after the economiser. Draught inside the kiln hood is indicated on a gauge, and other instruments indicate the temperature of the air entering the mills and air flow through the coolers. There are ammeters for all equipment.

A series of lights on the instrument board indicates how fast the tube-mill coal-feed motor is running, and is a positive indication of how the tube-mill coal level is being maintained. The different lights go on and off as the motor speed varies and, as the rate of feed is always under adjustment according to the mill load, the lights keep changing within the limits the feeder motor is rotating. Should the top light come on, or the bottom one, indicating extremes, a horn will sound and an adjustment will be made to the table feeder. When any of several horn signals on the board are shut off, the instrument board will flash a light to show the operator what piece of equipment caused the alarm to sound.

The foregoing notes are abstracted from an article by Mr. Bror Nordberg in "Rock Products," for December, 1941, in which many illustrations are given.

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